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(54) Title: DETERGENT–PACKAGE COMBINATION

(57) Abstract

A combination comprises at least one tablet of compacted particulate detergent composition, stored for at least 24 hours, within a packaging system having a moisture vapour transmission rate of less than 20 $g/m^2/24$ hours. A process of preparing the combination is disclosed.

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# DETERGENT - PACKAGE COMBINATION

## Introduction

The invention relates to a detergent package combination, and in particular to a detergent tablet - package combination.

One of the most prominent problems associated with detergent tablets is the provision of a tablet which is sufficiently strong to withstand processing, packaging, transport and handling without breaking or chipping, yet still be able to distintegrate quickly in an aqueous environment.

Detergent - package combinations are described in, for example, WO95 02681, which relates to a combination of a granular detergent composition which is contained in a packaging system having a moisture vapour transmission rate, hereinafter MVTR, of less than 20 $g/m^2/24$ hours.

WO 95/18215 describes a detergent article comprising a detergent mass of at least 100 g, typically 2 Kg to 5 Kg, having a barrier layer coating to prevent absorbtion of water. The teaching of this document is directed towards cast blocks of detergent material which, if unprotected, absorb large quantities of water rendering the blocks soft and malleable.

The present invention is directed towards the provision of an improved detergent-package combination which provides improved physical and functional

characteristics, for the detergent contained within the package.

## Statement of Invention

According to the invention, there is a provided a combination of at least one tablet of compacted particulate detergent composition stored for at least 24 hours within a closed packaging system having a moisture vapour transmission rate of less than $20g/m^2/24$ hours.

Surprisingly, it has been found that the above combination, when stored, results in a detergent tablet having improved physical characteristics, that is to say, the physical strength of the tablet increases considerably upon storage. This is a surprising and unexpected result. Even more surprising is the observation that, while the physical strength of the tablets increased, the dissolution time of the tablets in water actually decreases. Furthermore, the functional characteristics of the tablets have been found to be excellent.

Generally, the MVTR of the packaging system is less than 10 $g/m^2/24$ hours, ideally approximately 5 $g/m^2/24$ hours.

In a preferred embodiment of the invention, the detergent composition comprises either (a) a sodium tri-polyphosphate composition, or (b) a sodium acetate trihydrate composition optionally together with sodium citrate dihydrate. These components have been found to be highly effective disintegrants which, while allowing the tablets to be compressed to produce a tablet of

increased strength, allow a rapid disintegration of the tablet in water. Ideally, the detergent composition comprises phase I sodium tripolyphosphate which has been found to be a particularly effective disintegrant.

In a particularly preferred embodiment of the invention, the detergent composition comprises at least one tablet of a compacted particulate composition wherein the tablet or a region thereof comprises from 2% to 35% by weight of a salt which is sodium acetate trihydrate, potassium acetate or mixture thereof, optionally together with sodium citrate dihydrate, such that the total quantity of sodium acetate trihydrate, potassium acetate and sodium citrate dihydrate is from 7% to 50% by weight of the tablet or region thereof. Optionally the tablet may contain 15% to 93% of a water softening agent.

Alternatively, the detergent composition comprises a tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the particles, wherein the sodium tripolyphosphate in said particles contains water of hydration distributed throughout the tripolyphosphate in an amount between 1% and 5% by weight of the sodium tripolyphosphate in the particles. Ideally the detergent composition is stored in the packaging system for at least 1 week, preferably at least 2 weeks, more preferably at least 3 weeks, ideally at least 4 weeks. In one particularly preferred

embodiment of the invention the packaged detergent composition is stored for up to 8 weeks, and often for even greater than 8 weeks.

The packaging system will advantageously have a reasonable oxygen permeability, ideally not greater than 2000 $cm^3/m^3/24$ hours.

Typically, the packaging system comprises a material having a bulk density of less than $40g/m^2$.

Ideally, the packaging system comprises a polymeric film, preferably an oriented polypropylene film. Suitable films are sold under the trademark BICOR.

Alternatively, the packaging system may comprise a PET/PE laminate, preferably having a thickness of approximately 12 micron PET/40 micron PE.

In a preferred embodiment of the invention the detergent composition, is wrapped in a flow-wrap sealed polymer-based packaging system such as those described above.

Suitable packaging systems are the films sold under the trade name BICOR having the following codes:- MB 668, MB 666, MB 600, MB 450, MB 400, MD 447, MH 648, MW 648, MH 647 and MW 647.

The invention also provides a process for preparing a packaged detergent composition comprising the steps of:-

- placing at least one tablet of compacted particulate detergent composition in a packaging system having a MVTR of less than 20 $g/m^2/24$ hours;

- sealing the packaging system; and

- storing the thus formed sealed packaged detergent composition for at least 24 hours.

In one embodiment of the invention, the tablets comprise particulate detergent composition compacted with a force of at least 3N, typically greater than 4N, ideally greater than 5N and most preferably greater than 6N.

Ideally, a ratio of the DFS (KPa) to the $T_{90}$(mins) for the tablets is greater than 1, ideally greater than 2.

**Detailed Description**

The invention will be more clearly understood from the following description of some embodiments thereof given by way of example only.

**Example 1**

In this example of the invention, the combination comprises two sodium tripolyphospohate containing detergent tablets flow wrapped in sealed oriented polypropylene wrapping of the type sold under the trade mark BICOR MB 668.30, and stored for different periods of time under specific conditions of temperature and humidity.

A. Detergent Tablet Details

The composition and manufacture of the tablets is given below.

Tablets for use in fabric washing were made, starting with a spray-dried base powder of the following composition:

| | |
|---|---|
| Sodium linear alkylbenzene sulphonate | 11.83% |
| Sodium tripolyphosphate, type 1A [1] | 44.83% |
| Nonionic detergent [2] | 7.88% |
| Sodium silicate | 11.83% |
| Soap | 1.13% |
| Sodium carboxymethyl cellulose | 0.9% |
| Acrylate/maleate copolymer | 3.2% |
| Sodium sulphate and minor ingredients | 3.0% |
| Water | balance to 100% |

[1] This contained less than 30% of the phase I form of anhydrous sodium tripolyphoshate.

[2] $C_{13-15}$ fatty alcohol 7EO.

This powder was mixed with particles of sodium tripolyphosphate specified to contain 70% phase I form and contain 3.5% water hydration (Rhodia-Phos HPA 3.5 available from Rhone-Poulenc) and other detergent ingredients as tabulated below. As a comparative composition the base powder was mixed with urea and other detergent ingredients.

Two compositions thus contained the following percentages by weight.

| | Example 1 | Comparative A |
|---|---|---|
| Base Powder | 63.25 | 63.25 |
| Sodium perborate tetrahydrate | 10.4 | 10.4 |
| TAED granules | 4.0 | 4.0 |
| Anti-foam granule | 2.0 | 2.0 |
| Enzymes | 0.85 | 0.85 |
| Phosphonate | 0.5 | 0.5 |
| Sodium carbonate | 3.6 | 3.6 |
| HPA tripolyphosphate | 15.0 | --- |
| Urea | --- | 15.0 |

35g portions of each composition were made into cylindrical tablets of 44mm diameter, using a Carver hand press.

B. Packaging Details

The packaging system comprised a sealed flow-wrap oriented polypropylene system sold under the trade mark BICOR MB 668.30. The properties of this material are given in Table 1.A below:-

Table 1.A

| PROPERTIES | UNITS | MB 668 | | | |
|---|---|---|---|---|---|
| | | 20 | 25 | 30 | 40 |
| Tensile strength | $N/mm^2$ | 155 | 155 | 155 | 155 |
| Modulus of Elasticity | $N/mm^2$ | 2200 | 2200 | 2200 | 2200 |
| Breaking elongation | % | 175 | 175 | 175 | 175 |
| Coefficient of friction | | 0.25 | 0.25 | 0.25 | 0.25 |
| Water vapour permeability | $g/m^2/24\ h$ | 7.0 | 5.0 | 5.0 | 3.5 |
| Oxygen permeability | $cm^3/m^2/24$ | 1000 | 850 | 750 | 600 |
| Haze | % | 1.1 | 1.2 | 1.2 | 1.3 |
| Gloss | % | 85 | 85 | 85 | 85 |
| Heat seal range | 70 | 70 | 70 | 70 | |
| Seal strength | g/25mm | 400 | 400 | 400 | 400 |
| Dimensional stability | % | -5 | -5 | -5 | -5 |
| Unit weight | $g/m^2$ | 18.2 | 22.7 | 28.2 | 37.3 |
| Yield | $m^2/kg$ | 54.9 | 44.0 | 35.5 | 26.8 |

C. Comparative Results

The improved physical and functional characteristics of tablets of the combination according to the invention were tested. Two sets of detergent tablets manufactured according to the protocol described above were stored under the same conditions of temperature and relative humidity (37°C/70% RH), one of the sets of tablets being left open to the environment (open conditions), the other being packaged and stored according to the invention in the material described above (closed conditions). The strength, dissolution characteristics and functional stability were measured at 0, 1, 2, 4 and 8 weeks for tablets stored under open and closed conditions. The results are given in Table 1.B below.

The strength of these tablets was measured using an Instron universal testing machine to compress a tablet until fracture. The value of diametral fracture stress (DFS) was then calculated using the equation.

$$\frac{\sigma = 2P}{\Pi\ Dt}$$

where $\sigma$ is the diametral fracture stress in Pascals, P is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres.

The break-up, dispersion and dissolution of tablets was measured by a test procedure in which a tablet is placed on a plastic sieve with 2 mm mesh size which was immersed in 9 litres of demineralised water at ambient temperature

of 22°C and rotated at 200 rpm. The water conductivity was monitored until it reached a constant value.

The time for break up and dispersion of the tables was taken as the time ($T_{90}$) for change in the water conductivity to reach 90% of its final magnitude. This was also confirmed by visual observation of the material remaining on the rotating sieve.

**Table 1.B**

| Time | DFS (KPa) Closed Conditions | DFS (KPa) Open Conditions | Dissolution ($T_{90}$) Closed Conditions | Dissolution ($T_{90}$) Open Conditions | % Total TAED Closed Conditions | % Total TAED Open Conditions |
|---|---|---|---|---|---|---|
| 0 | 9.9 | 9.9 | 4.7 | 4.7 | 100 | 100 |
| 1 Week | - | - | - | 3.5 | - | - |
| 2 Week | 15.9 | 12.7 | 3.4 | 2.4 | 100 | - |
| 4 Week | 20.1 | 11.5 | 3.0 | 2.8 | 84 | 80 |
| 8 Week | 24.6 | - | 3.2 | - | 80 | - |

Referring to Table 1B, it is clear that tablets stored under closed conditions increase in strength during the storage period. This effect is also observed in tablets stored under open conditions however to a much lesser degree. Indeed at 4 week storage, the measured DFS for tablets stored according to the invention is almost twice that of the comparative example.

The tablets stored under closed conditions dissolved faster the longer they are stored. This effect is also observed for tablets stored under open conditions, as would be expected. However, comparing the ratio of Tablet Straight (DFS) to Dissolution Time ($T_{90}$) for tablets stored for 4 weeks under open and closed conditions the following results are obtained.

$DFS/T_{90}$ for open conditions $\simeq$ 3.8
$DFS/T_{90}$ for closed conditions $\simeq$ 6.7

It is clear from these results that the tablet/packaging combination of the present invention results in a tablet of improved dissolvability and strength. The stability of the TAED is slightly better in the tablets packed and stores according to the invention. Overall, the results clearly demonstrate the improved physical and functional characteristics inherent in packing and storing tablets according to the invention.

**Example 2**

In this example, the same combination is used as that of example 1 with the exception that the detergent

composition comprises a sodium acetate trihydrate composition made according to the protocol below.

Detergent Tablets Details

Tablets for use in fabric washing were made, starting with a base powder of the following composition:

| | |
|---|---|
| Coconut alkyl sulphate [1] | 2.9% |
| Zeolite A24 [2] | 52.9% |
| Sodium carbonate | 0.7% |
| Nonionic detergent [3] | 25.9% |
| Soap | 5.9% |
| Sodium carboxymethyl cellulose | 1.4% |
| Fluorescer | 0.4% |
| Acrylate/maleate copolymer | 0.7% |

1. The coconut alkyl sulphate was incorporated as preformed granules containing 45% coconut alkyl sulphate, 35% zeolite, 11% sodium carbonate, balance water and other salts.

2. Maximum aluminium zeolite P from Crosfields.

3. $C_{13-15}$ fatty alcohol 7EO.

This powder was mixed with sodium acetate trihydrate (from Merck as used in Example 1) and other detergent ingredients as tabulated below. As a comparative composition the base powder was mixed with sodium citrate dihydrate and other detergent ingredients and then sprayed with polyethylene glycol (Molecular Weight 1500) at 80°C.

The two compositions thus contained:

| | A (with Na-acetate 3aq). parts by weight | B (comparative) parts by weight |
|---|---|---|
| Base powder | 53.02 | 53.02 |
| Na-perborate 4aq. | 19.99 | 19.99 |
| TAED granules | 4.49 | 4.49 |
| Anti-foam granule | 3.42 | 3.42 |
| Enzymes | 1.5 | 1.5 |
| Phosphonate | 1.0 | 1.0 |
| Perfume | 0.43 | 0.43 |
| Na-acetate 3aq. | 16.13 | |
| Silicate-carbonate co-granule | | 5.5 |
| Na-citrate 2aq. | | 8.03 |
| PEG 1500 | | 2.5 |

35g portions of each composition were made into cylindrical tablets of 44 mm diameter, using a Carver hand press with various levels of compaction force.

The strength of these tablets was measured using an Instron universal testing machine to compress a tablet until fracture. The value of diametral fracture stress (DFS) was then calculated using the equation

$$\text{DFS} = \frac{2P}{\pi Dt}$$

where DFS is the diametral fracture stress in Pascals, P is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres.

The break-up, and dispersion of tablets was measured by the procedure of Example 1, using one tablet on the rotating sieve.

The results are set out in the following table:

| Compaction Force (kN) | A Tablets with Acetate.$3H_2O$ | | B Comparative tablets with citrate and PEG | |
|---|---|---|---|---|
| | Strength (DFS in kPa) | $T_{90}$ (minutes) | Strength (DFS in kPa) | $T_{90}$ (minutes) |
| 1 | 5.1 | 4.0 | -- | -- |
| 2 | 7.2 | 3.8 | 19.3 | 11.1 |
| 4 | 13.7 | 3.9 | 31 | 25 |
| 5 | 20.8 | 7.5 | 43 | 30 |

It can be seen that the tablets containing acetate trihydrate, made with 5kN compaction force were almost equal in strength to the comparative tablets made at 2kN force, but dispersed faster and did not require a process step of spraying polymer onto the powder.

Comparative Test

Tablet manufactured according to the protocol above were stored and tested under the test conditions set out in Example 1.

The test results are given in Table 2.

**Table 2.A**

| Time | DFS (KPa) Closed Conditions | DFS (KPa) Open Conditions | Dissolution ($T_{90}$) Closed Conditions | Dissolution ($T_{90}$) Open Conditions | % Total TAED Closed Conditions | % Total TAED Open Conditions |
|---|---|---|---|---|---|---|
| 0 | 18.4 | 18.4 | 3.1 | 3.1 | 100 | 100 |
| 1 Week | 19.5 | 10.0 | 3.1 | 5.0 | 95.6 | 43.4 |
| 2 Week | 20.5 | - | 3.1 | - | 82.6 | - |
| 4 Week | 18.3 | - | 2.8 | - | 91.3 | - |

Referring to Table 2B, and in particular to the figures on tablet strength, it is clear that tablets packed and stored according to the invention demonstrate an increase in strength over time when compared to the initial (T=O) measurements. This is not observed with tablets stored in open conditions. Comparing the dissolution times, as the strength of the tablets packed and stored according to the invention under closed conditions increases, the dissolution time remains relatively constant, and even decreases slightly after four weeks storage. The TAED stability of tablets packed and stored according to the ivention is excellent.

Further tests were carried out on the tablets to measure the maximum load (F Max) the tablets can sustain and the energy required (Break Enery) to break the tablet. The tests were carried out on freshly made tablets and tablets stored for 1, 4 and 6 days according to the invention. The results are given in Table 2.B below:

**Table 2.B**

| Time | F Max (N) | Break Energy (mJ) |
|---|---|---|
| T = 0 | 48.1 | 10.7 |
| 1 day | 53.5 | 10.4 |
| 4 days | 65.7 | 13.6 |
| 6 days | 57.3 | 12.9 |

The above results clearly show that the tablet combination according to the invention, even whens tored

for a period of 24 hours, results in tablets which are stronger than prior to storage.

The invention is not limited to the embodiments hereinbefore described which may be varied in both construction and detail.

CLAIMS

1. The combination of at least one tablet of compacted particulate detergent composition, stored for at least 24 hours within a closed packaging system having a moisture vapour transmission rate of less than 20 $g/m^2/24$ hours.

2. The combination of claim 1 in which the detergent composition comprises a sodium tri-polyphosphate composition.

3. The combination of claim 1 in which the detergent composition or a region thereof comprises particles whcih contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration distributed throughout the tripolyphosphate in an amount between 1% and 5% by weight of the sodium tripolyphosphate in the particles.

4. The combination of claim 1 or 2 in which the detergent composition comprises a sodium acetate trihydrate composition, optionally together with sodium citrate dihydrate.

5. The combination of claim 1 in which the detergent composition or a region thereof comprising 15% to 93% by weight of a water-softening agent characterised in that the tablet or a region thereof

contains 2% to 35% by weight of a salt which is sodium acetate trihydrate, potassium acetate or mixture thereof, optionally together with sodium citrate dihydrate, such that the total quantity of sidum acetate trihydrate, potassium acetate and sodium citrate dihydrate is from 7% to 50% by weight of the tablet or region thereof.

6. The combination of any preceeding claim wherein the detergent composition is stored in the packaging system for at least 1 week.

7. The combination of any of claims 1 to 3 wherein the detergent composition is stored in the packaging system for at least 2 weeks.

8. The combination of any preceeding claim in which the packaging system has an oxygen permeability not more than 2000 $cm^3/m^3/24$ hours.

9. The combination of any preceding claim in which the packaging system comprises a material housing a bulk density of less than 40 $g/m^2$.

10. The combination of any preceding claim in which the packaging system comprises a polymeric film.

11. The combination as claimed in claim 10 in which the packaging system comprises an oriented polypropylene film.

12. The combination of any of claims 1 to 10 in which the polymeric film comprises a PET/PE laminate.

13. The combination of claim 12 in which the PET/PE laminate thickness is approximately 12 micron PET/40 PE.

14. The combination of any preceding claim in which the detergent composition is wrapped in a flow-wrap sealed polymer-based packaging system.

15. The combination substantially as hereinbefore described with reference to the accompanying examples.

16. A process for preparing a packaged detergent composition comprising the steps of:-

- placing at least on tablet of compacted particulate detergent composition in a packaging system having a moisture vapour transmission rate of less than 20 $g/m^2/24$ hours;
- sealing the packaging system; and
- storing the thus formed sealed packaged detergent composition for at least 24 hours.

# INTERNATIONAL SEARCH REPORT

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PCT/EP 98/01274

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D17/00 C11D17/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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[X] Further documents are listed in the continuation of box C.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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